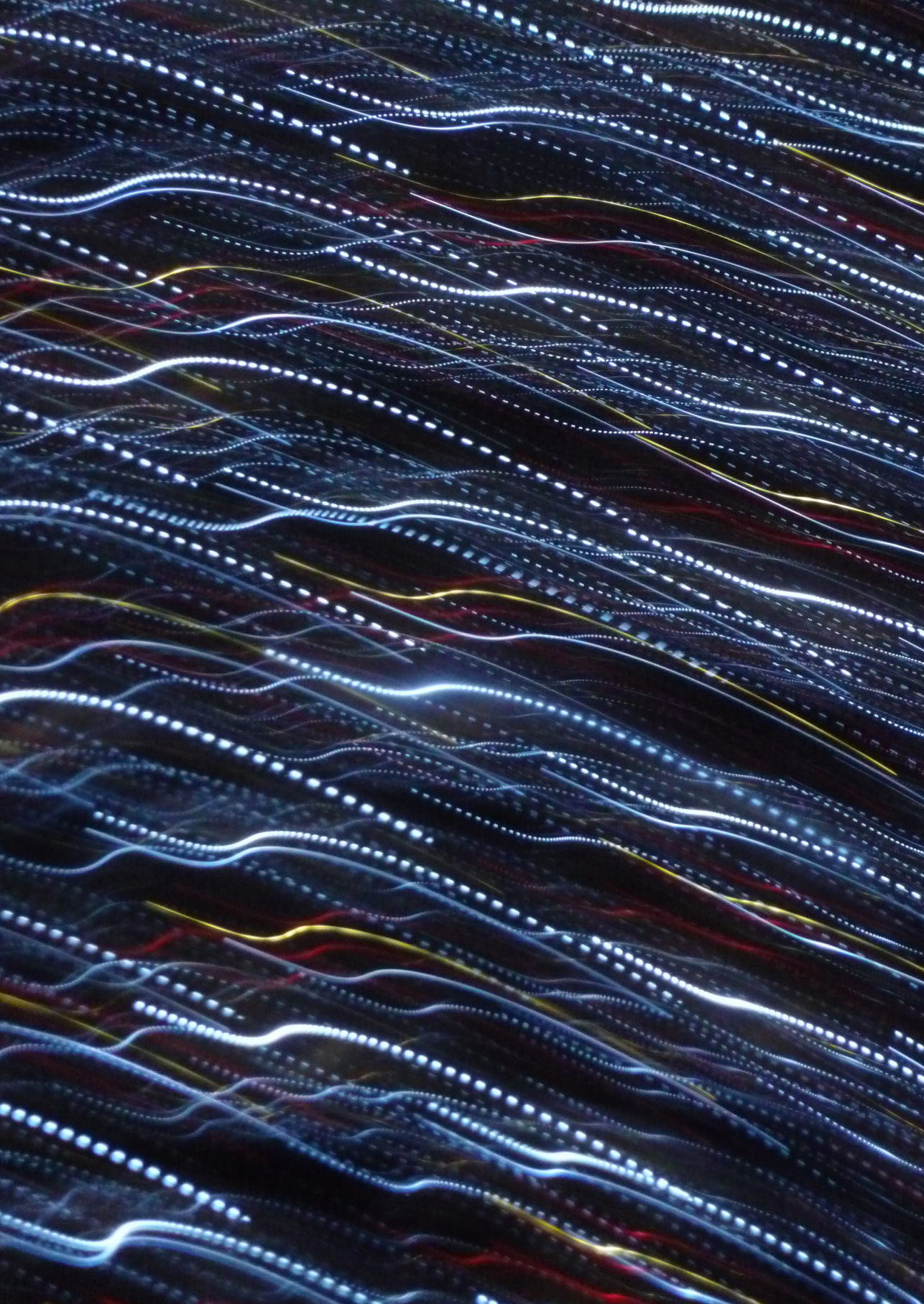
Donor-Acceptor Stenhouse Adducts

Lerch, Michael Markus

A



When I started doing chemistry, I did it the way I fished - for the excitement, the discovery, the adventure, for going after the most elusive catch imaginable in uncharted seas.

Karl Barry Sharpless
Nobel lecture, 2001

Appendix – A

Materials and Methods

A.1 Synthesis

General reagent information: Preparation of commercially unavailable compounds: unless stated otherwise, all reactions were carried out in oven- and flame-dried glassware using standard Schlenk techniques and were run under nitrogen atmosphere. The reaction progress was monitored by Thin-layer chromatography (TLC). Starting materials, reagents and solvents were purchased from *Sigma–Aldrich*, *Acros*, *Fluka*, *Fischer*, *TCI*, *J.T. Baker* or *Macron* and were used as received, unless stated otherwise. Solvents for the reactions were of quality puriss., p.a.. Anhydrous solvents were purified by passage through solvent purification columns[1] (MBraun SPS-800). For aqueous solutions, deionized water was used.

General considerations: Thin-layer chromatography analyses were performed on commercial Kieselgel 60 F_{254} silica gel plates with fluorescence-indicator UV_{254} (*Merck, TLC silica gel 60 F_{254}*). For detection of components, UV light at $\lambda = 254$ nm or $\lambda = 365$ nm was used. Alternatively, oxidative staining using aqueous basic potassium permanganate solution ($KMnO_4$) or aqueous acidic cerium phosphomolybdic acid solution (Seebach's stain[2]) was employed. Drying of solutions was performed with $MgSO_4$ and volatiles were removed with a rotary evaporator (at 50 °C water bath temperature).

General analytical information: Nuclear Magnetic Resonance spectra were measured with an Agilent Technologies 400-MR (400/54 Premium Shielded) spectrometer (400 MHz) or an Agilent Technologies Innova 500 NMR spectrometer (500 MHz). All spectra were measured at room temperature (22–24 °C). Chemical shifts for the specific NMR spectra were reported relative to the residual solvent peak [in ppm; $CDCl_3$: $\delta_H = 7.26$; $CDCl_3$: $\delta_C = 77.16$; CD_2Cl_2: $\delta_H = 5.32$; CD_2Cl_2: $\delta_C = 53.84$; DMSO-d_6: $\delta_H = 2.50$; DMSO-d_6: $\delta_C = 39.52$; CD_3CN: $\delta_H = 1.94$; CD_3CN: $\delta_C = 1.32, 118.26$; d_6-acetone: $\delta_H = 2.05$; d_6-acetone: $\delta_C = 29.84, 206.26$; toluene-d_8: $\delta_H = 2.08, 6.97, 7.01, 7.09$; toluene-$d_8$: $\delta_C = 137.48, 128.87, 127.96, 125.13, 20.43$; D_2O: $\delta_H = 4.79$; CD_3OD: $\delta_H = 3.31$; CD_3OD: $\delta_C = 49.00$].[3] The multiplicities of the signals are denoted by *s* (singlet), *d* (doublet), *t* (triplet), *q* (quartet), *m* (multiplet), *br* (broad signal), *app* (apparent). All ^{13}C-NMR spectra are ^1H-broadband decoupled.

High-resolution mass spectrometric measurements were performed using a Thermo scientific LTQ OrbitrapXL spectrometer with ESI ionization. The molecule-ion M^+, $[M + H]^+$ and $[M - X]^+$, respectively, are given in *m/z*-units. Melting points were recorded using a Stuart analogue capillary melting point SMP11 apparatus.

A.2 UV/visible static and steady state measurements

For spectroscopic measurements, solutions in Uvasol® grade solvents were measured in a 10 mm quartz cuvette, unless stated otherwise. UV/vis absorption spectra were recorded on an Agilent 8453 UV/vis, a Hewlet-Packard HP 8543 diode array or an Analytik Jena Specord S600 diode array. Temperature-control was exerted through a Peltier based temperature controlled cuvette holder (QuantumNorthwest). A non-coherent white light-source was purchased from Thorlabs (OSL1-EC). In chlorinated solvents, an optical cut-off filter (< 440 nm, SCF-50S-44Y) was used. The obtained UV/vis spectra were baseline corrected. Data-analysis was performed using Origin, Prism, R (*https://www.r-project.org/*) or Spectragryph software.

A.3 ^{1}H-NMR *in situ*-irradiation measurements

For *in situ*-irradiation experiments, an LED-based irradiation setup was built according to a reported system.[4] The fiber-optic cable (*M28L05; Ø400 µm, 0.39 NA, SMA-SMA Fiber Patch Cable, 5 Meters*) and the LEDs were purchased from Thorlabs: 470 nm Fiber-coupled LED (*M470F3, 21.8 mW, FWHM = 20 nm*); 530 nm Fiber-coupled LED (*M530F2, 9.6 mW, FWHM = 30 nm*). NMR tubes were purchased from Wilmad-LabGlass (SP Scienceware): *WGS-5BL, Coaxial Insert for 5 mm NMR Sample Tube and 535-PP-7, 5 mm Thin Wall Precision NMR Sample Tube 7" L, 600MHz*. Spectra were measured with an Agilent Technologies Inova 500 spectrometer (500 MHz).

A.4 Light sources

Sources for irradiation have been purchased from commercial suppliers. Technical details (λ_{em} or λ_{max}, intensity, type and supplier) are summarized in Table A1 and A2.

Table A1 | Comparison of used commercial light sources (Chapter 2–6).

Entry	λ_{max} (nm)	Type	Supplier
1	312	ENB-280C/FE	Spectroline
2	365 (br.)	ENB-280C/FE	Spectroline
3	365	M365F1	ThorLabs
4	370	MARL 260019 UV EMITTER, TO-46, 100DEG	Farnell
5	430	607-4304H6	Mouser
6	590	604-APTD1608SYC/J3	Mouser
7	640	604-APTD1608SEC/J3	Mouser
8	white light (wl)	OSL1-EC	ThorLabs
8	white light (halogen)	Plusline ES Small 160W, 3100 lm	Philips

A.4.1 Fiber-coupled LEDs

Table A2 | Used fiber-coupled LEDs for NMR *in situ*-irradiation (Chapters 3 and 5) were purchased from ThorLabs and used with a fiber optic cable (*M28L05; Ø400 µm, 0.39 NA, SMA-SMA Fiber Patch Cable, 5 Meters*).

Entry	λ_{max} (nm)	FWHM (nm)	Intensity (mW)	Type
1	470	20	21.8	M470F3
2	530	30	9.6	M530F2

A.4.2 Optical Filters

The 440 nm optical cut-off filter was purchased from OptoSigma (Molenaar Optics; SCF-50S-44Y, transmittance limit: λ_T = 440±5 nm, wavelength slope width $\Delta\lambda$ < 25 nm).

Table A3 | Used optical band-pass filters were purchased from Andover Corporation and used in combination with the high-intensity white light source (OSL1-EC).

Entry	λ_{center} (nm)	Bandwidth (nm)	Transmission (%)	Type
1	430.0 +3/-0	10.0 +2/-2	45	430FS10-50
2	546.1 +2/-0	10.0 +2/-2	55	546FS10-50
3	577.0 +2/-0	10.0 +2/-2	55	577FS10-50

A.5 References

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Appendix – B

Short Biography

Michael M. Lerch
February, 15th 1989
Lucerne, Switzerland

michi.lerch@gmail.com

Education

Nov. 2016 – Jan. 2017	Research stay at University of Florence/LENS Laserlab Europe (5 weeks; Prof. Dr. Paolo Foggi; Dr. Mariangela Di Donato; LENS002289; *"Understanding the Photoswitching Mechanism of Donor-Acceptor Stenhouse Adducts"*
Jul. 2014 – Jul. 2018	**PhD Candidate**, Rijksuniversiteit Groningen, Research group of Prof. Dr. Ben L. Feringa (Nobel Prize 2016) PhD project (supramolecular chemistry, photochemistry) *"Emerging Photoswitches: Controlling Biological Function with Light"*
Sep. 2013 – Apr. 2014	Visiting Student Researcher at California Institute of Technology Master's Thesis, Prof. Dr. Robert H. Grubbs (Nobel Prize 2005)/Prof. Dr. Erick M. Carreira
Sep. 2012 – Jun. 2014	**MSc Interdisciplinary Sciences**, Major in Biology and Chemistry, ETH Zürich Master's Thesis (synthetic organic methodology): *"Inductive Effects in Wacker-Type Oxidations of Internal Alkenes"*
Jan. 2012 – Jun. 2012	ERASMUS LLP exchange semester at University of Cambridge Courses and Bachelor's Thesis
Sep. 2009 – Sep. 2012	**BSc Interdisciplinary Sciences**, biochemical – physical pathway, ETH Zurich Bachelor's Thesis (total synthesis) Prof. Dr. Steven V. Ley/Prof. Dr. Erick M. Carreira: *"Synthesis and Utility of the Sidechain Precursor for the Total Synthesis of Spirangiens A and B"*

Appendix – C

List of Publications

ORCID: *http://orcid.org/0000-0003-1765-0301*

Original Research Articles:

Michael M. Lerch,† Mariangela Di Donato,† Adèle D. Laurent, Miroslav Medveď Alessandro Iagatti, Laura Bussotti, Andrea Lapini, Wybren Jan Buma, Paolo Foggi, Wiktor Szymański,* Ben L. Feringa*
(† contributed equally)
Angew. Chem. Int. Ed. **2018**, DOI: *10.1002/anie.201803058*
Angew. Chem. **2018**, DOI: *10.1002/ange.201803058*
"Solvent Effects on the Actinic Step of Donor-Acceptor Stenhouse Adduct Photoswitching"

Michael M. Lerch, Miroslav Medveď, Andrea Lapini, Adèle D. Laurent, Alessandro Iagatti, Laura Bussotti, Wiktor Szymański, Wybren Jan Buma, Paolo Foggi, Mariangela Di Donato,* Ben L. Feringa*,
J. Phys. Chem. A **2018**, *122* (4), 955–964
DOI: *10.1021/acs.jpca.7b10255*
"Tailoring Photoisomerization Pathways in Donor-Acceptor Stenhouse Adducts: The Role of the Hydroxyl Group"

Mariangela Di Donato,† Michael M. Lerch,† Andra Lapini, Adèle D. Laurent, Alessandro Iagatti, Laura Bussotti, Svante P. Ihrig, Miroslav Medveď, Denis Jacquemin, Wiktor Szymański, Wybren Jan Buma, Paolo Foggi, Ben L. Feringa*
(† contributed equally)
J. Am. Chem. Soc. **2017**, *139* (44), 15596–15599
DOI: *10.1021/jacs.7b09081*
"Shedding Light on the Photo-Isomerization Pathway of Donor–Acceptor Stenhouse Adducts"

Mickel J. Hansen, Michael M. Lerch, Wiktor Szymański*, Ben L. Feringa*
Angew. Chem. Int. Ed. **2016**, *55* (43), 13514–13518; DOI: *10.1002/anie.201607529*
Angew. Chem. **2016**, *128* (43), 13712–13716; DOI: *10.1002/ange.201607529*
"Direct and Versatile Synthesis of Red-shifted Azobenzenes"

Michael M. Lerch, Mickel J. Hansen, Willem A. Velema, Wiktor Szymański*, Ben L. Feringa*
Nat. Commun. **2016**, *7*, 12054
DOI: *10.1038/ncomms12054*
"Orthogonal Photoswitching in a Multifunctional Molecular System"

Michael M. Lerch, Sander J. Wezenberg, Wiktor Szymański, Ben L. Feringa*
J. Am. Chem. Soc. **2016**, *138* (20), 6344–6347
DOI: *10.1021/jacs.6b01722*
"Unraveling the Photoswitching Mechanism in Donor–Acceptor Stenhouse Adducts"

Willem A. Velema,† Mickel J. Hansen,† Michael M. Lerch, Arnold J. M. Driessen, Wiktor Szymański, Ben L. Feringa*
(† contributed equally)
Bioconjug. Chem. **2015**, *26* (12), 2592–2597
DOI: *10.1021/acs.bioconjchem.5b00591*
"Ciprofloxacin–Photoswitch Conjugates: A Facile Strategy for Photopharmacology"

Michael M. Lerch, Bill Morandi, Zachary K. Wickens, Robert H. Grubbs*
Angew. Chem. Int. Ed. **2014**, *53* (33), 8654–8658; DOI: *10.1002/anie.201404712*
Angew. Chem. **2014**, *126* (33), 8798–8802; DOI: *10.1002/ange.201404712*
"Rapid Access to β-Trifluoromethyl-Substituted Ketones: Harnessing Inductive Effects in Wacker-Type Oxidations of Internal Alkenes"

Reviews:

Michael M. Lerch, Wiktor Szymański,* Ben L. Feringa*
Chem. Soc. Rev. **2018**, *47*, 1910-1937
DOI: *10.1039/C7CS00772H*
"The (Photo)chemistry of Stenhouse Photoswitches: Guiding Principles and System Design"

Michael M. Lerch,† Mickel J. Hansen,† Gooitzen M. van Dam, Wiktor Szymański,* Ben L. Feringa*
(† contributed equally)
Angew. Chem. Int. Ed. **2016**, *55* (37), 10978–10999; DOI: *10.1002/anie.201601931*
Angew. Chem. **2016**, *128* (37), 11140–11163; DOI: *10.1002/ange.201601931*
"Emerging Targets in Photopharmacology"

Mickel J. Hansen, Willem A. Velema, Michael M. Lerch, Wiktor Szymański,* Ben L. Feringa*
Chem. Soc. Rev. **2015**, *44* (11), 3358–3377
DOI: *10.1039/c5cs00118h*
"Wavelength-Selective Cleavage of Photoprotecting Groups: Strategies and Applications in Dynamic Systems"

Patents:

Bill Morandi, Zachary K. Wickens, Robert H. Grubbs, Michael M. Lerch *"Process for the Synthesis of Ketones from Internal Alkenes"* **US20140194604**, filed 08.01.14, granted 04.08.2015.

Appendix – D

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Pursuing a PhD can be a transformative experience. Doing research is not always easy, but neither is life. What research means to me is the joy of going out there and having fun exploring thoughts and concepts and discovering things that might be self-evident for most people but are fascinating to me. I have been lucky to be surrounded by people that allowed me to do exactly this in the past four years. At the end of my PhD, I am left to express my gratefulness to all the people I have met and that have influenced me.

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